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METHOD FOR DETERMINATION OF SODIUM AND POTASSIUM (FLAME PHOTOMETRIC)

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METHOD FOR DETERMINATION OF SODIUM AND POTASSIUM (FLAME PHOTOMETRIC)

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(*Continued on page* 2)

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Indian Standard

METHOD FOR DETERMINATION OF SODIUM AND POTASSIUM (FLAME PHOTOMETRIC)

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 April 1980, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 When a solution containing salts of sodium or potassium is atomized into a gas flame, light characteristic of these elements is emitted, the intensity being a function of concentration. Salts of many other elements cause similar emission. The flame photometer consists of apparatus for giving a reproducible amount of emitted light for a given concentration of element in the test solution, and for determining the intensity of such emission as a function of concentration of the element without excessive interference from other emitted light.

0.3 For a given element light of wavelength other than that characteristic of the element to be determined must be eleminated before accurate intensity measurements can be made. This is accomplished by selective filtration of the emitted light, by use of interference filters, or by a suitable slit arrangement in light-dispersing devices such as prism or grating monochromators.

0.4 The flame photometric technique is now being increasingly employed in analytical work for determination of micro-quantities, the determination of sodium and potassium can also be accomplished by atomic absorption spectrophotometric method, which is not covered in this standard.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes method for the estimation of sodium and potassium by flame photometer.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS :1070-1977*) shall be employed in tests.

> NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. OUTLINE OF THE METHOD

3.1 The material is powdered and homogenized so as to pass through 150 micron IS Sieve. A known mass of the air dried sample is suitably decomposed in acid mixture, filtered and diluted to a definite volume. This test solution or its suitable dilutions are then aspirated in the flame photometer. From the galvanometer reading the concentration of sodium or potassium can be obtained by referring to a calibration curve. Ground water sample or its suitable dilution can be directly aspirated and alkali metals determined.

4. APPARATUS

4.1 Flame Photometer

5. REAGENTS

5.1 Concentrated Hydrochloric Acid — *See* IS : 265-1976†.

5.2 Concentrated Nitric Acid — *See* IS : 264-1976‡.

5.3 Hydrofluoric Acid

5.4 Potassium Chloride

5.5 Sodium Chloride

6. PROCEDURE

6.1 Preparation of Standard Sodium Solution — Weigh accurately 2·542 2 g of sodium chloride previously dried at 120°C to constant mass

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*Specification for water for general laboratory use (*second revision*).

†Specification for hydrochloric acid (*second revision*).

‡Specification for nitric acid (*second revision*).

and cooled to room temperature in a desiccator over silica gel. Transfer the weighed salt with water to a 250-ml beaker, dissolve in about 100 ml of water and add 10 ml of concentrated hydrochloric acid. Transfer the solution quantitatively to one-litre one-mark volumetric flask and dilute to the mark. Mix well and store in a polyethylene bottle. This solution contains 1 mg/ml of sodium.

6.1.1 *Preparation of the Standard Matching Solutions* — Into a 100-ml volumetric flask take 10 ml of the standard solution (*see* **6.1**), add 2 ml concentrated hydrochloric acid and make up the volume. This solution will contain 100 µg/ml of sodium. Dilute 1, 2, 3, 4 and 5 ml of this solution to 100 ml in 100-ml volumetric flasks. Number these flasks as 1, 2, 3, 4 and 5 respectively. The strength of these solutions shall be as given in Table 1. Store these solutions in polyethylene bottles.

TABLE 1 STRENGTH OF SODIUM SOLUTIONS FOR CALIBRATION

NUMBER OF SOLUTION (1)	VOLUME OF STANDARD SOLUTION ml (2)	QUANTITY OF SODIUM µg/ml (3)
1	1·0	1·0
2	2·0	2·0
3	3·0	3·0
4	4·0	4·0
5	5·0	5·0

6.2 Preparation of Standard Potassium Solution — Weigh accurately 1·906 8 g of potassium chloride dried at 400°C and cooled in a desiccator. Transfer to a 250-ml beaker with washing. Dissolve in about 100 ml of water. Add 10 ml of concentrated hydrochloric acid. Transfer to 1-litre volumetric flask and dilute to mark. Mix well and store in polyethylene bottle. The solution contains 1 mg/ml of potassium.

6.2.1 *Preparation of the Standard Matching Solutions* — Into a 100-ml volumetric flask pipette out 10 ml of the standard solution (*see* **6.2**). Add 2 ml of concentrated hydrochloric acid and dilute to mark. This solution will contain 100 µg/ml of potassium. Dilute 1, 2, 3, 4 and 5 ml of this solution to 100 ml in 100-ml volumetric flasks. Number these flasks as 1, 2, 3, 4 and 5 respectively. The strength of these solutions shall be as given in Table 2. Store these solutions in polyethylene bottles.

TABLE 2 STRENGTH OF POTASSIUM SOLUTIONS FOR CALIBRATION

NUMBER OF SOLUTION (1)	VOLUME OF STANDARD SOLUTION ml (2)	QUANTITY OF POTASSIUM µg/ml (3)
1	2·0	2·0
2	4·0	4·0
3	6·0	6·0
4	8·0	8·0
5	10·0	10·0

6.3 Preparation of Calibration Chart

6.3.1 Switch on the flame photometer in advance to allow sufficient time for its stabilization. Put sodium/potassium filter in position, adjust sensitivity of the apparatus and galvanometer should read zero while atomizing water with 2 percent hydrochloric acid.

6.3.2 Atomize the standard matching solution (5 µg/ml for sodium and 10µg/ml for potassium) into the flame and adjust sensitivity to read 100 points on digital or full scale on galvanometer. Check zero and 100. Atomize the rest of standard solutions (*see* **6.1.1** and **6.2.1**) and measure the intensities of emitted radiation from galvanometer readings.

NOTE — Quantity of solution atomized in the flame should be kept constant per unit of time throughout preparation of calibration curve, as well as sample analysis, by keeping the pressure constant.

6.3.3 Plot a curve having the number of micrograms of sodium and potassium contained in the standard matching solutions on the abscissa and the corresponding values of the intensities of radiation from galvanometer reading on the ordinate.

NOTE — For complex materials standard matching solutions should also contain equivalent amounts of major interfering constituents.

6.4 Determination

6.4.1 *Preparation of Test Solution* — Powder the material so that it passes 150 micron IS Sieve [*see* IS : 460 (Part I) - 1978* and IS : 460 (Part II) - 1978†]. Weigh accurately about 5 g of the powdered material, dry in an air oven at 110°C for an hour, cool in a desiccator and weigh to constant mass. The difference will give moisture content of the sample. Decompose a suitable mass of the test sample to bring sodium and potassium into solution as chloride/nitrate. For oresand minerals proceed as follows.

6.4.1.1 Weigh accurately 0·2 g of as received mineral sample and transfer to a 100-ml beaker. Add 5 ml of concentrated hydrochloric acid, 5 ml of concentrated nitric acid and 10 ml of water. Boil for 30 to 40 minutes till reaction is complete. Evaporate to syrupy consistency, extract the residue with hydrochloric acid (2 percent), filter if necessary, transfer quantitatively to a 100-ml volumetric flask, transfer the washings and make up the volume to the mark.

6.4.1.2 Decompose the silicates (0·2 g) with hydrofluoric acid (5 ml) and nitric acid (10 ml) in a 50-ml platinum dish and evaporate just to dryness or to syrupy consistency. Repeat the process thrice for complete decomposition. Add 10 ml of nitric acid and evaporate again. Transfer quantitatively into a 100-ml beaker with minimum of water. Add 10 ml of hydrochloric acid. Evaporate to syrupy consistency. Take up with 2 ml of concentrated hydrochloric acid and 10 ml of water. Transfer solution and washings quantitatively to a 100-ml volumetric flask and make up to the mark.

6.4.1.3 For determination of alkali metals in organic materials incinerate at 400 to 450°C the sample, previously dried and pulverised to 150-micron IS Sieve, till carbon is completely burnt. Then proceed as in **6.4.1.2**.

6.4.2 Aspirate the solution. If galvanometer readings exceed maximum value in the standard curve (*see* **6.3**), dilute it suitably, add 2 ml of hydrochloric acid per 100 ml and aspirate. A reagent blank should be run throughout the estimation. Check the standard curve (**6.3.3**) before or while aspirating the test solution.

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*Specification for test sieves : Part I Wire cloth test sieves (*second revision*).

†Specification for test sieves : Part II Perforated plate test sieves (*second revision*).

7. CALCULATION

7.1 Concentration of alkali metal, percent by mass

$$= \frac{A \times D}{10\,000}$$

where

A = concentration in µg/ml of alkali metal in the sample solution, and

D = dilution factor for the sample taken.